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COMPLETE SPECIFICATION

NO DRAWINGS

Process for the Preparation of Organophilic Oxides and Chromate Pigments

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This invention relates to the preparation of organophilic pigments.

It is usual to treat lithopone pigments with water-soluble soaps in order to improve their mixing and dispersing properties. Lithopone, which is known to be a mixture of zinc sulfide and barium sulfate, can be thus made organophilic to a satisfactory extent. The same process is, however, not suitable for oxide pigments, since on the addition of the soap solution they will form a highly dispersed suspension. Oxide pigments do not precipitate and thus cannot be processed for industrial use. Chromate pigments change their color upon the addition of the soap, whereby their quality is deteriorated.

We have found that the above drawbacks may be removed by treating the pigment substance with anionic surface active agent, and subsequently acidifying the solution, preferably to pH-5. Thereupon the oxide pigments become readily filterable, and easily removable from the liquid. After washing,—if necessary—they are dried and, if desired, disintegrated. After acidifying, chromate pigments recover their original color so that excellent quality organophilic and readily filterable pigments are obtained. The organophilic pigments thus obtained are highly dispersible in organic solvents and can be

readily processed to stable suspensions.

According to the invention there is provided a process for rendering pigments selected from oxide, hydrated oxide and chromate pigments organophilic which comprises reacting an aqueous suspension of the pigment with a solution of an anionic surface active agent, acidifying the solution, separating the pigment from the liquid, then drying and, if desired, disintegrating the product thus obtained.

Mineral acids, e.g. hydrochloric acid, sulphuric acid and phosphoric acid, are preferably used to acidify the aqueous medium; however, strong organic acids, e.g. acetic acid may also be used.

Suitable anionic surface active agents for use in the process according to the invention include alkali metal and ammonium salts of saturated or unsaturated fatty acids having at least 8 carbon atoms, preferably having from 16 to 18 carbon atoms. Other surface active agents which may be used are the alkali metal or ammonium salts of fatty acid containing an OH group, for example ricinoleic acid. There may also be used alkali metal or ammonium salts of partly polymerised unsaturated fatty acids as well as alkali metal and ammonium salts of industrial stearine, comprising a mixture of stearic, palmitic and oleic acids.

Another class of surface active agents which may be employed as anionic surface active agents are the alkali metal salts of alkyl sulphonics, aryl sulphonics and mixed alkyl-aryl sulphonics acids.

The pigments used in the process according to the invention are oxide or hydrated oxide pigments precipitated or prepared by pyrogenic processes, for example titanium dioxide, red and yellow iron oxides, green chromium oxide, red bauxite (obtained by heating bauxite) and aluminium oxide and

chromate pigments, for example zinc chromate, zinc tetroxy chromate, lead chromate and basic lead chromate. These pigments have positive surface charges in aqueous solution and are capable of ion exchange when reacted with anionic surface active agents. The surface active agent ions adsorbed on the surface of the pigment particles become irreversibly bound to the surface upon subsequent acidification and drying following upon filtering. Thus, an organophilic film fixed on the surface of the said pigment particles is produced, whereby the dry pigment particles exhibit an organophilic character.

In the case of chromate pigments the optimum amount of the substance rendering them organophilic can be established by experimental trials. It has been found that the amount depends on the kind and particle size of the pigment. It is advisable to carry out the trials in the following manner: A series of equal amounts of pigment are suspended in equal amounts of water, then equal volumes of aqueous solutions are added containing increasing amounts of the anionic surface active agents. The suspensions are thoroughly agitated, and after a certain time the volume of the sediment is ascertained. The largest amount of sediment, that is, the amount of the surface active agent associated therewith will indicate the optimum amount. The pigments treated with the amount so established will exhibit the best organophilic properties and will unite most readily and with the highest dispersion with the organic substance of the paint vehicles where they form non-settling stable suspensions.

The step of rendering the pigment substances organophilic can be most conveniently effected as follows: 10 to 50% pigment is suspended in water, and a 1 to 4% aqueous solution, heated preferably to 70 to 80°C, of the surface active agent is added thereto at an elevated temperature, preferably at 70 to 80°C. 0.5 to 5% of the organophilizing agent, depending on the experimental trial, based on the pigment weight, is used. The pigment suspension so prepared is then acidified to pH 5, then the pigments are readily separated from the water, washed, dried preferably at 60 to 100°C and disintegrated.

Example 1.

500 g titanium dioxide are suspended in 2 litres of distilled water, and 1 litre 1% ammonium stearate is added while stirring. Stirring is carried on, and diluted hydrochloric acid is used for adjusting the pH to 5. The pigment suspension thereupon forms a readily filterable precipitate, which is filtered off, washed if necessary with water, dried at 80 to 100°C, then granulated in an edge runner. The pigment so obtained is once milled in a three-roller mill and is used for preparing an enamel paint which is still entirely

free from sediments after one year. Subjected to a weather exposure test, it showed a life thrice as long as an enamel paint containing non-organophilic pigments. It has better spreading properties and a higher gloss and the films obtained therefrom are highly water-proof and the pigment particles will not migrate to the surface.

Organophilic pigments can be produced in the same manner from iron oxide yellow and iron oxide red, chrome oxide green, aluminium oxide and bauxite red.

Example 2.

Experimental trials showed that the best addition to a lead chromate pigment in an aqueous suspension is 1% by weight of ammonium stearate, as the latter produced the largest sediment volume. 500 g lead chromate are suspended in 2 litres of water, then 500 g of 1% ammonium stearate are added at 65 to 70°C under brisk agitation. Thereupon the light yellow pigment turns orange. The pH of the pigment suspension is adjusted with hydrochloric acid to 5, whereupon the chrome yellow regains its original colour. The precipitate is filtered, washed with water, dried at 60 to 100°C and homogenised in an edge runner. The ammonium salts of fatty acids have the advantage over sodium soaps that upon acidification the original colour shade can more preferably be reestablished. The NH_4Cl which is formed can be driven off in course of drying.

Organophilic pigments can be prepared likewise from zinc chromate, zinc tetrahydroxy chromate, barium chromate, or strontium chromate.

Example 3.

The organophilic pigment obtained in accordance with Example 1 or 2 can be readily employed in non-polar or slightly polar organic media and lacquer base materials, for example oil varnish or oleoresinous varnish, solutions of epoxy resins esterified with fatty acids, oil modified alkyd resin solutions.

Organophilic pigments of good wetting properties which can be suspended in polar organic media, such as alcohols, ketones, esters, nitrocellulose lacquers, spirit varnishes, alkyd-urea-formaldehyde vehicles, and alkyd melamine-formaldehyde vehicles can be obtained with the process described in the preceding examples, but using, instead of ammonium stearate, the equivalent amount of surface active agent derived from a fatty acid containing an OH-group, for example ammonium ricinoleate.

WHAT WE CLAIM IS:—

1. A process for rendering pigments selected from oxides, hydrated oxide and chromate pigments organophilic which comprises reacting an aqueous suspension of the pigment with a solution of an anionic surface active agent, acidifying the solution, separating the pigment from the liquid, then

- drying and, if desired disintegrating the product thus obtained.
2. A process according to claim 1, wherein the aqueous solution is acidified to pH 5.
- 5 3. A process according to claim 1 or claim 2, wherein a mineral acid is used for the acidification.
4. A process according to any of claims 1 to 3, wherein the anionic surface active agent is an alkali metal or ammonium salt of a fatty acid having at least 8 carbon atoms.
- 10 5. A process according to claim 5, wherein the fatty acid has from 16 to 18 carbon atoms.
- 15 6. A process according to claims 1 to 3, wherein the anionic surface active agent is an alkali metal or ammonium salt of a fatty acid containing an OH group.
7. A process according to claim 6 wherein 20 in said fatty acid is ricinoleic acid.
8. A process according to any of claims 1 to 3, wherein the anionic surface active agent is an alkali metal or ammonium salt of industrial stearine.
9. A process according to any of claims 1 to 3, wherein the anionic surface active agent is an alkali metal or ammonium salt of a partly polymerised unsaturated fatty acid.
10. A process according to any of claims 1 to 3 wherein the anionic surface active agent is an alkali metal salt of an alkyl sulphonic acid, aryl sulphonic acid or mixed alkyl-aryl sulphonic acid.
11. A process for the preparation of organophilic pigments substantially as herein described with reference to any of the Examples.
12. Organophilic pigments whenever prepared by a process as claimed in any of the preceding claims.

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